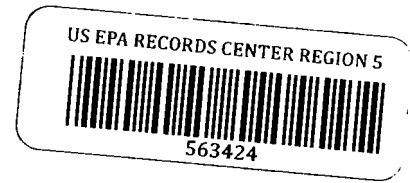


#4603

QUALITY ASSURANCE PROJECT PLAN  
ADDENDUM IV



Behr Dayton Thermal System VOC  
Plume Site  
Montgomery County, Dayton, Ohio  
Remedial Investigation/Feasibility Study  
WA No. 138-RICO-B5FH/Contract No. EP-S5-06-01

*Prepared for*



January 2016

**ch2m:**

QUALITY ASSURANCE PROJECT PLAN (QAPP) ADDENDUM IV  
Remedial Investigation/Feasibility Study  
BEHR DAYTON THERMAL SYSTEM VOC PLUME SITE  
MONTGOMERY COUNTY, DAYTON, OHIO  
WA No. 138-RICO-B5FH/Contract EP-S5-06-01

Lead Organization: United States Environmental Protection Agency (EPA)

Prepared by: CH2M HILL (CH2M)

Date: January 2016

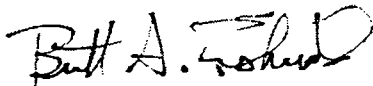
Approved by:

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EPA, Region 5, Remedial Project Manager  
Erik Hardin

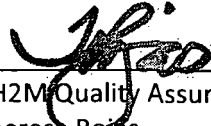
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**QAPP documents written for previous site work:**

<b>Title</b>	<b>Approval Date</b>
<i>Quality Assurance Project Plan, Behr Dayton Thermal System VOC Plume Site, Montgomery County, Dayton, Ohio, Remedial Investigation/Feasibility Study, WA No. 046-RICO-B5FH/Contract No. EP-S5-06-01</i>	June 2010
<i>Quality Assurance Project Plan Amendment I, Behr Dayton Thermal System VOC Plume Site, Montgomery County, Dayton, Ohio, Remedial Investigation/Feasibility Study, WA No. 138-RICO-B5FH/Contract No. EP-S5-06-01</i>	June 2014
<i>Quality Assurance Project Plan Addendum II, Behr Dayton Thermal System VOC Plume Site, Montgomery County, Dayton, Ohio, Remedial Investigation/Feasibility Study, WA No. 138-RICO-B5FH/Contract No. EP-S5-06-01</i>	November 2014
<i>Quality Assurance Project Plan Addendum III, Behr Dayton Thermal System VOC Plume Site, Montgomery County, Dayton, Ohio, Remedial Investigation/Feasibility Study, WA No. 138-RICO-B5FH/Contract No. EP-S5-06-01</i>	February 2015

# Introduction

This addendum is a supplement to the Behr Dayton Thermal System Volatile Organic Compound (VOC) Plume Site quality assurance project plan (QAPP) (June 2010), QAPP Addendum I (June 2014), QAPP Addendum II (November 2014), and QAPP Addendum III (February 2015). Unless otherwise indicated in the addendum, the data quality objectives, sampling rationale, procedures, analysis, and validation will follow the procedures outlined in the original QAPP, Addendum I, Addendum II, and Addendum III.

Remedial investigation activities were performed from 2011 through 2015 in accordance with the specified procedures identified in the approved QAPP and subsequent addendums. Results of the groundwater and soil samples collected as part of the remedial investigation indicated that additional information is needed to further delineate the extent of the VOC plume in groundwater, and delineate the extent of VOCs in soils at the Gem City Chemicals Facility (Gem City). Additionally, geophysical soil data from Gem City is needed to support the feasibility study (FS).

This addendum includes the following activities that were not previously included in the original QAPP or subsequent addendums: the collection of groundwater grab samples, installation of new monitoring wells, sampling of new and existing monitoring wells, and collection of soil samples from the Gem City Chemicals Facility (Gem City). This addendum describes the investigation activities and includes methods and requirements to perform the additional investigation activities described above. A new field operating procedure (FOP) is included for groundwater grab sampling, and laboratory standard operating procedures (SOPs) are included for analyses to be performed by the subcontract laboratory.

The FOP for sample collection to be performed as part of this addendum includes the following:

- *Groundwater Grab Sample Collection*, FOP #11 (Attachment 1)

Sample analysis will be performed by CT Laboratories in Baraboo, Wisconsin. Additional analytical methods to be performed under this addendum include the following:

- VOCs in groundwater by EPA SW-846 Method 8260B (Attachment 2)
- Total organic carbon in soil by Lloyd Kahn/SW-846 9060A (Attachment 2)
- Dry/bulk density for soil samples (Attachment 2)
- Total solids for soil samples (Attachment 2)
- Grain size for soil samples (Attachment 2)

Geophysical soil samples will be considered informational data only for further development of the conceptual site model and will not be evaluated against project action levels or validated. VOC data from groundwater and soil samples will be validated in accordance with the previously approved QAPP (CH2M 2010).

## 1.1 Sampling Process Design and Rationale

### 1.1.1 Groundwater Grab Sampling

An anticipated 25 soil borings will be completed at locations shown in Figures 1 through 3 using a sonic drill rig for continuous soil coring to the bottom of each boring. After advancing the borehole to the targeted depth, one groundwater grab sample will be collected from each sample interval listed in Table 1. Groundwater grab samples will be generally collected from the shallow (approximately 25 to 30 feet below ground surface [bgs]), intermediate (approximately 45 to 50 feet bgs), and the deep (approximately 75 to 80 feet bgs) zones of the upper aquifer. The sampling locations are assumed and may be adjusted once access agreements are established, utilities are evaluated, and pending changes in lithology are observed during drilling activities.



**Table 1. Sampling Intervals**

*Quality Assurance Project Plan Addendum IV*

*Behr Dayton Thermal System VOC Plume Site, Montgomery County, Dayton, Ohio*

Targeted Sample Zone	Number of Sampling Locations	Approximate Boring and Sample Depths (feet bgs)
Shallow	9	25-30
Intermediate	10	45-50
Deep	6	75-80

bgs = below ground surface

Samples will be analyzed by the subcontracted laboratory for VOCs on a 24-hour turnaround time. Analytical results of each groundwater grab sample will be presented to EPA upon receipt from the laboratory to determine if additional sampling locations may be required or if proposed sampling locations may be eliminated during this investigation. The location and number of borings and samples may be adjusted throughout the project based on the results of samples collected at the direction of EPA. The results of the groundwater grab samples will be used to determine the approximate extents of the VOC plume, as defined by EPA maximum contaminant levels<sup>1</sup> (MCLs), in groundwater in areas where the boundaries are currently undefined.

In accordance with FOP #11 (Attachment 1), *Groundwater Grab Sample Collection*, groundwater grab samples will be collected using a decontaminated stainless-steel screen sampler packed at the top of the sample interval and a decontaminated submersible sample pump. All water added to the borehole during drilling will be evacuated prior to collecting a groundwater grab sample. Groundwater grab samples will be collected using a decontaminated submersible sample pump (stainless-steel Monsöon/Hurricane or equivalent). Following completion of sample collection, each borehole will be grouted from total depth to the surface in accordance with the Ohio EPA *Technical Guidance Manual for Ground Water Investigations*, Chapter 9, "Sealing Abandoned Monitoring Wells and Boreholes."<sup>2</sup>

Groundwater grab samples will be collected by CH2M staff from an estimated 25 sample intervals (see Figures 1 through 3 and Worksheet #18). The sampling locations were reviewed and agreed upon by EPA on December 17, 2015, and Ohio EPA on December 20, 2015. The groundwater samples will be submitted to subcontracted laboratory, CT laboratories, for VOC analysis by method SW-846 8260B (Attachment 2) with an expedited 24-hour turnaround time for preliminary data. Quality assurance (QA)/quality control (QC) samples will be collected in accordance with the previously approved QAPP (CH2M 2010).

### 1.1.2 Groundwater Monitoring Well Installation

Preliminary results of the groundwater grab samples will be received by CH2M, and the data will be provided to the EPA work assignment manager. EPA and CH2M will review the preliminary data, which will be used to determine if monitoring wells should be installed during this supplemental investigation. The final number and location of monitoring wells will be determined by EPA. If monitoring wells are to be installed at the direction of EPA, they will be installed in the shallow zone and intermediate zones of the aquifer as identified in Figures 1 and 2. Monitoring wells will not be installed in the deep zone of the upper aquifer during this event.

<sup>1</sup> EPA. National Primary Drinking Water Regulations. Available online: <http://www.epa.gov/dwstandardsregulations>. Accessed January 14, 2016.

<sup>2</sup> Ohio EPA. 2009. *Technical Guidance Manual for Ground Water Investigations*, Chapter 9, "Sealing Abandoned Monitoring Wells and Boreholes." February. Available online: [http://epa.ohio.gov/Portals/28/documents/TGM-09\\_1009.pdf](http://epa.ohio.gov/Portals/28/documents/TGM-09_1009.pdf). Accessed January 14, 2016.

It is currently anticipated that two to six monitoring wells will be installed during this supplemental investigation. The following monitoring wells are planned to be installed:

- Two shallow monitoring wells installed to an approximate depth of 30 feet bgs in the northern area of the site.
- Two potential shallow monitoring wells installed to an approximate depth of 30 feet bgs in the southwestern area of the site.
- Two potential intermediate monitoring wells installed to an approximate depth of 55 feet bgs in the southern area of the site.

Boreholes will be drilled with a minimum 6-inch-diameter hole to allow for the installation of a 2-inch-diameter monitoring well and a minimum 2-inch annular space surrounding the well casing. Monitoring wells will be installed following FOP #1, *Monitoring Well Installation*, in the previously approved QAPP (CH2M 2010). Following installation of the monitoring wells, each new monitoring well will be developed following FOP #2, *Monitoring Well Development*, in the QAPP (CH2M 2010).

### 1.1.3 Groundwater Sampling

Following monitoring well installation, groundwater samples will be collected from up to eight monitoring wells for the purpose of delineating the VOC plume in groundwater. The sampling network will consist of up to six monitoring wells anticipated to be installed during this supplemental investigation, and two existing wells located on the edge of the VOC plume (see Figure 4 and Worksheet #18).

Groundwater samples are currently planned to be collected from the following monitoring wells:

- Up to four shallow monitoring wells to be installed to an anticipated depth of 30 feet bgs
- Up to two intermediate monitoring wells to be installed to an anticipated depth of 55 feet bgs
- Existing shallow monitoring wells MW-201-S and MW-202 S, both located in the southwestern area of the site

Monitoring wells will be sampled by CH2M staff following FOP #3, *Well Inspection, Water Level Collection, and Groundwater Sampling*, in the previously approved QAPP (CH2M 2010). Following sample collection, the groundwater samples will be shipped to CT Laboratories for analysis of VOCs following Method SW-846 8260B (Attachment 2). VOC samples collected from monitoring wells will be submitted for analysis on a 21-day turnaround time. QA/QC samples will be collected in accordance with the previously approved QAPP (CH2M 2010).

### 1.1.4 Soil Sampling at the Gem City Chemicals Facility

Soil samples will be collected from 10 locations within the Gem City property for the primary purpose of delineating VOCs observed in soils during the 2014 and 2015 remedial investigation sampling events and to support data needs for the FS report. Soil samples will be collected from continuously cored boreholes through use of a Geoprobe direct-push technology following FOP #5, *Soil Sample Collection*, in the previously approved QAPP (CH2M 2010).

Soil samples will be collected for VOC analysis from the 0- to 0.5-foot bgs and 4- to 6-foot bgs interval at each location to delineate exceedances of the industrial regional screening level (RSL) identified during the 2014 and 2015 sampling events. At 3 of the 10 soil borings, one soil sample will be collected for VOC analysis from the 8- to 10-foot bgs interval to delineate exceedances of the industrial RSL observed in soil boring GC-SB05 in 2014 (Figure 5). At 2 of the 10 soil borings, soil samples will be collected from the 13- to 15-foot bgs interval to vertically delineate elevated VOC concentrations observed in the 8- to 10-foot bgs intervals from soil borings GC-SB103 and GC-SB105 collected in 2014. Locations of the soil

borings and sample depths are presented in Figure 5. Analysis of VOCs in soil samples will be performed by the subcontracted laboratory following Method SW-846 8260B in the previously approved QAPP Addendum III (CH2M 2015).

Surface soil samples at Gem City collected during the 2014 and 2015 remedial investigation had matrix interference issues for VOC analysis. Site history indicates that coal was stored on this portion of the site, which may be causing the interference. In addition to the collection of soil samples for VOC analysis, 7 soil samples will be collected from surface intervals for analysis of total organic carbon (TOC) following Method Lloyd Kahn/SW-846-9060A by CT Laboratories (Attachment 2). TOC is not a contaminant of concern at the site and will not be screened against any project action levels.

Geophysical parameters will be collected from two locations to support further development of the conceptual site model and to support the development of remedial alternatives during the FS. At the two boring locations, GC-SB110 and GC-SB114 (Figure 5), an initial soil boring will be installed to approximately 15 feet bgs to classify the soil lithology and collect the designated soil samples for VOC analysis. An offset boring will be installed approximately 1 foot away from the original soil boring for the purpose of collecting intact soil cores with a Shelby tube for analysis of geophysical parameters. If two distinct types of soils are observed in the soil boring, such as predominately clay or sand layers, one 2-foot intact soil sample will be collected from each soil type. If soil is primarily homogeneous, intact soil samples will be collected from the 2- to 4-foot bgs and 7- to 9-foot bgs depth intervals. Intact soil samples will be sent to CT Laboratories for analysis of TOC, dry/bulk density (bulk density-core method), moisture content (SM2540B&G/SW-846 8000C), and grain size (sieve). The analytical SOPs are provided in Attachment 2.

A summary of soil samples and analysis is presented in Table 2. QA/QC samples will be collected in conjunction with VOC samples in accordance with the QAPP. Additional QA/QC samples will not be collected for geophysical parameters or TOC analyses.

**Table 2. Gem City Soil Sampling Summary**  
*Quality Assurance Project Plan Addendum IV*  
*Behr Dayton Thermal System VOC Plume Site, Montgomery County, Dayton, Ohio*

Soil Sample Location	Sample Depth (feet bgs)	Required Analysis
GC-SB108	0-0.5	VOCs and TOC
	4-6	VOCs
GC-SB109	0-0.5	VOCs and TOC
	4-6	VOCs
	8-10	VOCs
GC-SB110	0-0.5	VOCs
	4-6	VOCs
	8-10	VOCs
	13-15	VOCs
Two intact soil cores for dry/bulk density, moisture content, TOC, and grain size		

**Table 2. Gem City Soil Sampling Summary**  
*Quality Assurance Project Plan Addendum IV*  
*Behr Dayton Thermal System VOC Plume Site, Montgomery County, Dayton, Ohio*

Soil Sample Location	Sample Depth (feet bgs)	Required Analysis
GC-SB111	0-0.5	VOCs
	4-6	VOCs
	8-10	VOCs
GC-SB112	0-0.5	VOCs and TOC
	4-6	VOCs
GC-SB113	0-0.5	VOCs and TOC
	4-6	VOCs
GC-SB114	0-0.5	VOCs
	4-6	VOCs
	8-10	VOCs
	13-15	VOCs
Two intact soil cores for dry/bulk density, moisture content, TOC, and grain size		
GC-SB115	0-0.5	VOCs and TOC
	4-6	VOCs
GC-SB116	0-0.5	VOCs
	4-6	VOCs
GC-SB117	0-0.5	VOCs
	4-6	VOCs

### 1.1.5 Investigation-derived Waste

Investigation-derived waste (IDW) samples will be collected to characterize solid and aqueous waste generated during the investigation for offsite disposal. IDW samples will be collected and analyzed in accordance with the previously approved QAPP (CH2M 2010). It is assumed that two IDW soil samples will be collected to characterize solid waste generated during the drilling and installation of monitoring wells, and that two IDW water samples will be collected to characterize aqueous waste generated during drilling, development, and sampling of the monitoring wells. Analysis of waste characterization samples will be performed by the subcontract laboratory, CT Laboratory, and will include the following parameters: total or toxicity characteristic leaching procedure (TCLP) VOCs, total or TCLP semivolatile organic compounds, total or TCLP Resource Conservation and Recovery Act 8 metals, total polychlorinated biphenyls (PCBs), corrosivity, and ignitability.

## 1.2 Schedule

Groundwater grab sampling is anticipated to begin in January 2016, with monitoring wells being installed in January/February 2016. Groundwater sampling and soil sampling at the Gem City facility will occur following well installation during a separate mobilization in February/March 2016. Preliminary results for groundwater grab samples will be requested within 24 hours of laboratory receipt. Laboratory

analysis and reporting will be completed within 21 days of sample receipt by the laboratory. Upon receipt of analytical data, CH2M will validate the data and prepare a data quality evaluation report within 21 days, after receipt of data from the subcontracted laboratory. Soil samples collected for analysis of geophysical parameters and TOC will be considered informational data only and will not be validated.

## 1.3 Project Action Levels

Worksheet #15-4 presents the project action levels and laboratory limits. In addition to listing the particular analytes, screening levels, and limits, the table identifies where quantitation limits (QLs) or MDLs are greater than project action limits (PALs). Although the information was taken into consideration when planning analytical protocol for the site and could lead to some uncertainty, it does not prevent conclusions from being drawn with respect to the project objectives for the following reasons:

- Even though some QLs are greater than the respective screening levels, MDLs are closer to and could be less than the applicable PALs. The laboratory instrumentation would likely detect a constituent if present at a concentration greater than its MDL, and such a result would be reported as estimated because it is less than the QL.
- If a particular analyte has a QL or MDL greater than a screening level and there are sufficient other analytes in the same constituent group that would likely be detected with a QL or MDL less than the screening values, then determinations for further action at the site can be made with sufficient confidence.
- Standard EPA methods are proposed, which often have QLs below PALs due to the low target risk levels incorporated into the PALs.
- The analytes with PALs below the QLs are not expected to be related to releases from the site.

Worksheet #15-5 presents the geophysical parameters for soil samples that will be collected to further develop the conceptual site model and to support development of remedial alternatives during the FS. There are no project action levels for these parameters.

## 1.4 Analytical Worksheet Updates

The following worksheets are provided as a supplement to the worksheets in the QAPP and subsequent QAPP addendums.

# Sheet #12-12: Measurement Performance

Parameters and TOC<sup>a</sup>

Method/SOP <sup>b</sup>	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S & A)
P #7	Completeness	> 90% Laboratory analysis	Percent Completeness	S & A
P #7	Accuracy/bias	± % Recovery	LCS	A
P #7	Representativeness	Contamination of sample	MB	S & A
P #7	Comparability	Qualitative measure for field sampling procedures	LCS	A
P #7	Sensitivity	Evidence of shift in instrument response or zero setting MDLs	LCS	A

Parameters include the following parameters: TOC, dry/bulk density, total moisture, and grain size.  
 (CH2M 2010).

MB = method blank, FB = field blank, RPD = relative percent difference, MDL = method detection limit

# Sheet #15-4: Project Action Limits and Specific Detection/Quantitation Limits

CAS Number	Human Health Screening Level <sup>a</sup> (µg/L)	Human Health Screening Level Source	Achievable Laboratory Limits	
			Laboratory-specific Method Detection Limit <sup>f</sup> (µg/L)	Laboratory Reporting Limit (µg/L)
71-55-6	200	MCL	0.009	0.1
79-34-5	0.076	RSL	0.017	0.2
79-00-5	0.041	RSL	0.015	0.1
75-34-3	2.8	RSL	0.021	0.1
75-35-4	7	MCL	0.04	0.1
87-61-6	0.7	RSL	0.04	0.1
120-82-1	0.4	RSL	0.04	0.1
96-12-8	0.00033	RSL	0.07	0.2
106-93-4	0.0075	RSL	0.015	0.1
95-50-1	30	RSL	0.025	0.1
107-06-2	0.17	RSL	0.015	0.1
78-87-5	0.44	RSL	0.012	0.1
541-73-1	0.48	RSL	0.03	0.1
106-46-7	0.48	RSL	0.03	0.1
123-91-1	0.46	RSL	5	20
76-13-1	1,500	VISL	0.05	0.2



NDUM IV  
ME SITE, MONTGOMERY COUNTY, DAYTON, OHIO

78-93-3	560	RSL	0.5	1
591-78-6	3.8	RSL	0.12	1
108-10-1	630	RSL	0.18	1
67-64-1	1,400	RSL	1	2
71-43-2	0.46	RSL	0.008	0.1
74-97-5	8.3	RSL	0.03	0.1
75-27-4	0.13	RSL	0.016	0.1
75-25-2	3.3	RSL	0.019	0.1
74-83-9	0.75	RSL	0.038	0.1
75-15-0	81	RSL	0.07	0.2
56-23-5	0.41	VISL	0.018	0.1
108-90-7	7.8	RSL	0.03	0.1
75-00-3	2,100	RSL	0.07	0.2
67-66-3	0.22	RSL	0.01	0.1
74-87-3	19	RSL	0.026	0.1
156-59-2	3.6	RSL	0.022	0.1
10061-01-5	0.47	RSL	0.012	0.1
110-82-7	1,000	VISL	0.027	0.1
124-48-1	0.87	RSL	0.018	0.1
75-71-8	7.4	VISL	0.013	0.1
100-41-4	1.5	RSL	0.027	0.1
98-82-8	45	RSL	0.026	0.1
179601-23-1	19	RSL	0.06	0.2
79-20-9	2,000	RSL	0.1	0.2

EN1202151037MKE



1634-04-4	14	RSL	0.025	0.1
108-87-2	1,000	VISL	0.02	0.1
75-09-2	5	MCL	0.2	1
95-47-6	19	RSL	0.026	0.1
100-42-5	100	MCL	0.024	0.1
127-18-4	4.1	RSL	0.01	0.1
108-88-3	110	RSL	0.011	0.1
156-60-5	36	RSL	0.026	0.1
10061-02-6	0.47	RSL	0.014	0.1
79-01-6	0.28	RSL	0.02	0.1
75-69-4	520	RSL	0.022	0.2
75-01-4	0.019	RSL	0.019	0.1

value between the Maximum Contaminant Level (MCL), where available, the EPA Tap Water Regional Screening Level (RSL); or the Vapor are November 2015 values based on a target Excess Lifetime Cancer Risk (ELCR) =  $1 \times 10^{-6}$  and Hazard Index (HI) = 0.1; VISLs are based on EPA exposure scenario, a target ELCR =  $1 \times 10^{-6}$ , a HI = 1, and a default groundwater temperature = 25 degrees C. RSL values as referenced by (USEPA,

s used as a surrogate for trans-1,3-Dichloropropene

s used as a surrogate for cis-1,3-Dichloropropene.

s a surrogate for Methylcyclohexane.

s used as a surrogate for trans-1,3-Dichloropropene.

ated annually and are subject to change.

creening levels are lower than the laboratory method detection limit and/or quantitation limit. Refer to Section 1.3 for a discussion.

# Sheet #18: Sampling Locations and Methods

Matrix/Media	Sample Depth	Analytical Group	Number of Samples	Sampling SOP <sup>b</sup>
Groundwater Grab	Approximate shallow interval: 25-30 feet bgs	VOCs	9	FOP #11
Groundwater Grab	Approximate intermediate interval: 45-50 feet bgs	VOCs	10	FOP #11
Groundwater Grab	Approximate deep interval: 75-80 feet bgs	VOCs	6	FOP #11
Groundwater Grab / MS/MSD	--	VOCs	2/2	FOP #11
Groundwater/ Field Duplicate	--	VOCs	3	FOP #11
Equipment Rinsate Blank	--	VOCs	2 (1 per shift per type of sampling equipment)	FOP #11
Blank Water/ Trip Blank	--	VOCs	1 per cooler containing VOC samples	FOP #11
Samples				
Groundwater/ Monitoring Well	Approximate shallow interval: 30 feet bgs	VOCs	4	FOP #3
Groundwater/ Monitoring Well	Approximate intermediate interval: 55 feet bgs	VOCs	2	FOP #3
Groundwater/ MS/MSD	--	VOCs	1/1	FOP #3
Groundwater/ Field Duplicate	--	VOCs	1	FOP #3

Matrix/Media	Sample Depth	Analytical Group	Number of Samples	Sampling SOP <sup>b</sup>
Equipment Rinsate Blank	--	VOCs	2 (1 per shift per type of sampling equipment)	FOP #3
Blank Water/ Trip Blank	--	VOCs	1 per cooler containing VOC samples	FOP #3
Soil Boring/Soil	0-0.5 feet bgs	VOCs and TOC	10 (VOCs), 5 (TOC)	FOP #5
	4-6 feet bgs	VOCs	10	
	8-10 feet bgs	VOCs	3	
	13-15 feet bgs	VOCs	2	
	*Intact soil cores from sand and clay layers	Geophysical parameters and TOC	4	
Soil/ MS/MSD	--	VOCs	2/2	FOP #5
Soil / Field Duplicate	--	VOCs	3	FOP #5
Equipment Rinsate Blank	--	VOCs	2 (1 per shift per type of sampling equipment)	FOP #5

code will also be assigned to each sample.  
d QAPP (CH2M, 2010) and this QAPP Addendum.

atrix spike; MSD = matrix spike duplicate

# 

atory, CT Laboratories

is/certifications: National Environmental Laboratory Accreditation Conference

night Shipping

	Method/SOP	Container(s)	Preservation	Preparation and Analytical Holding Time	Preliminary Data Turnaround	Data Package Turnaround
ib)	SW-846 8260B	Three 40-mL VOA Vial	HCL, 4°C	14 Days	24 hours	21 days
	SW-846 8260B	Three 40-mL VOA Vial	HCL, 4°C	14 Days	21 days	21 days
	SW-846 8260B	5 g of soil in each of two 40-mL VOA vial DI water, one 40-mL vial methanol	4°C (DI water and/or methanol)	48 hours to freeze DI water vials/ 14 days to analysis	21 days	21 days
	Lloyd Kahn and SW-846-9060A	One 2-oz poly or Shelby Tube	4°C	28 days	21 days	21 days
	Bulk Density-Core	Shelby Tube	--	--	21 days	21 days
	SM2540B&G/ SW-846-8000C	Shelby Tube	4°C (recommended)	7 days	21 days	21 days
	Grain Size (Sieve)	Shelby Tube	--	--	21 days	21 days

l operating procedure; HCL = hydrochloric acid; DI = deionized water;

# Sheet #23: Analytical Standard Operating Procedures

File and Date	Definitive or Screening Data	Matrix	Analytical Group	Instrument/ Equipment Type	Laboratory Performing Analysis	Modified for Project?
Organic Compounds .5	Definitive	Ground-water	VOCs	GC/MS	CT Laboratory Baraboo, WI	No
in Soil, 1/6/2016	Screening	Soil	Total Organic Carbon	Carbonaceous Analyzer	CT Laboratory Baraboo, WI	Yes
Method,	Screening	Soil	Dry/Bulk Density	--	CT Laboratory Baraboo, WI	No
015 )	Screening	Soil	Total Moisture	--	CT Laboratory Baraboo, WI	No
5	Screening	Soil	Grain size	Sieves	CT Laboratory Baraboo, WI	No

## Figures























Attachment 1  
FOP #11

**BEHR DAYTON THERMAL GROUNDWATER PLUME SITE**  
**FIELD OPERATING PROCEDURE**  
**FOR GROUNDWATER GRAB SAMPLE COLLECTION**

**Prepared by:**

**Date**

12/18/15

*Peter Schuster*

Author

**Reviewed by:**

**Date**

12/18/15

*Bill A. Schuster*

Site Manager

**Approved by:**

**Date**

12/18/15

*Kimberly D. Schuster*

Project Quality Manager

## FIELD OPERATING PROCEDURE-11

# Groundwater Grab Sample Collection

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## I. Purpose

This Field Operating Procedure (FOP) provides a general guideline for the collection of groundwater samples using sonic drilling methods.

## II. Scope

This is a general description of groundwater grab sampling using a discrete groundwater grab sampler.

## III. Equipment and Materials

- Drill rig and sampling rods with slotted stainless steel screen and an inflatable packer assembly
- Polyethylene or Teflon™ sampling tubing, and stainless steel sample pump (e.g. Hurricane or Monsoon type pump) or a peristaltic pump.
- Water quality meter(s) (e.g. YSI, turbidity meter, etc.)
- Water level meter
- Pre-cleaned sample containers with appropriate preservative
- Clean nitrile gloves

## IV. Calibration

Calibrate water quality instruments in accordance with manufacturer specifications.

## V. Procedures and Guidelines

Groundwater grab samples are collected as a time expedient method when monitoring wells are not needed or where conditions are unknown. This FOP describes the basic procedure for groundwater grab sample collection.

1. Decontaminate downhole equipment in accordance with the QAPP.
2. Drill to the planned sampling depth and set the casing.
3. Using the drill rig, place the slotted reusable stainless steel screen at the bottom of the borehole pull back the downhole casing to expose the screen.
4. Insert the decontaminated submersible pump and new tubing through the rods or insert new peristaltic pump tubing through rods, depending on which method is used.

5. If multiple samples are being collected from the same borehole, deploy a packer to isolate the zone being sampled from the overlying zone. The packer assembly is attached above the stainless steel screen and remains in the outer casing to seal the annular space above the sample point.
6. If drilling fluids were added, the isolated zone will be purged of the volume of water added to the borehole during drilling at a rate of approximately 2 gallons per minute. Purging will then continue until the water is visibly clear of sediment or turbidity is less than 50 Nephelometric Turbidity Units (NTUs) or until an additional 20 gallons have been removed.
7. Record the groundwater quality parameters on the sampling forms (see records management section).
8. Reduce the pumping rate to a flow less than 0.5 liters per minute prior to sample collection. Monitor water levels in accordance with SOP-12.
9. Measure water quality parameters in accordance with SOP-12 (Groundwater Sampling), with the exception of turbidity. Purge until water quality parameters have stabilized in accordance with SOP-12. This information will be recorded on groundwater sampling forms.
10. Fill all sample containers in accordance with the QAPP.
11. Remove the downhole equipment, decontaminate the pump in accordance with the QAPP (if using a submersible pump), and discard the sample tubing.
12. Backfill the borehole in accordance with local, state, or federal regulations and repair the surface to match existing conditions, as required.

## **VI. Records Management**

Purging information will be recorded on a groundwater sampling form for each sample interval. Groundwater water quality parameters (oxidation-reduction potential (ORP), pH, specific conductance (SpC), turbidity, dissolved oxygen (DO) and temperature), as well as groundwater flow rate and depth to water will be recorded approximately every 5 minutes. Records will be uploaded to the project SharePoint site and retained in accordance with the contract.

## **VII. Quality Control and Quality Assurance**

- Document calibration of water quality instruments in the field book.
- Document the decontamination of non-disposable equipment prior to use in the field notebook.
- Verify source of drilling fluids, and the volume of drilling fluids added and purged is recorded in the field book.
- Prior to installation of the packer assemble, inspect the packer for damage and the

lead casing for damage or obstructions.

- If used, verify the isolation packer is properly installed by checking the pressure gauge and ensuring the pressure is stable, at 120psi. Check the pressure every 5 minutes throughout the process to ensure the packer is inflated.
- Verify the depth to water using a water level meter measured to the nearest 0.01 foot, prior to deploying the packer.
- Verify the borehole is abandoned in accordance with federal, state, or local regulations.
- Ensure that the sample has been collected and handled in accordance with the QAPP and applicable laboratory standard operating procedures (SOP's).

## **VIII. Attachments**

Groundwater Sampling Form.

## **IX. References**

Not applicable.



## Attachment 2

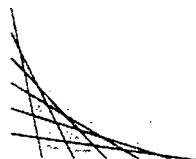
### Laboratory SOPs

# CT LABORATORIES

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**Test:** VOC 8260 LL  
**Method:** EPA 8260C  
**Matrix:** GROUND WATER

Analyte	CAS #	DL	LOD ug/L	LOQ/RL ug/L	
1,1,1-Trichloroethane	71-55-6	0.009	0.02	0.1	
1,1,2,2-Tetrachloroethane	79-34-5	0.017	0.02	0.2	
1,1,2-Trichloroethane	79-00-5	0.015	0.02	0.1	
1,1-Dichloroethane	75-34-3	0.021	0.05	0.1	
1,1-Dichloroethene	75-35-4	0.04	0.05	0.1	
1,2,3-Trichlorobenzene	87-61-6	0.04	0.05	0.1	
1,2,4-Trichlorobenzene	120-82-1	0.04	0.05	0.1	
1,2-Dibromo-3-chloropropane	96-12-8	0.07	0.1	0.2	
1,2-Dibromoethane	106-93-4	0.015	0.02	0.1	
1,2-Dichlorobenzene	95-50-1	0.025	0.05	0.1	
1,2-Dichloroethane	107-06-2	0.015	0.02	0.1	
1,2-Dichloropropane	78-87-5	0.012	0.02	0.1	
1,3-Dichlorobenzene	541-73-1	0.03	0.05	0.1	
1,4-Dichlorobenzene	106-46-7	0.03	0.05	0.1	
1,4-Dioxane	123-91-1	5	10	20	
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.05	0.1	0.2	
2-Butanone	78-93-3	0.5	0.5	1	
2-Hexanone	591-78-6	0.12	0.2	1	
4-Methyl-2-pentanone	108-10-1	0.18	0.2	1	
Acetone	67-64-1	1	1	2	
Benzene	71-43-2	0.008	0.02	0.1	
Bromochloromethane	74-97-5	0.03	0.05	0.1	
Bromodichloromethane	75-27-4	0.016	0.02	0.1	
Bromoform	75-25-2	0.019	0.02	0.1	
Bromomethane	74-83-9	0.038	0.05	0.1	
Carbon disulfide	75-15-0	0.07	0.1	0.2	
Carbon tetrachloride	56-23-5	0.018	0.02	0.1	
Chlorobenzene	108-90-7	0.03	0.05	0.1	
Chloroethane	75-00-3	0.07	0.1	0.2	
Chloroform	67-66-3	0.01	0.02	0.1	
Chloromethane	74-87-3	0.026	0.05	0.1	
cis-1,2-Dichloroethene	156-59-2	0.022	0.05	0.1	
cis-1,3-Dichloropropene	10061-01-5	0.012	0.02	0.1	
Cyclohexane	110-82-7	0.027	0.05	0.1	
Dibromochloromethane	124-48-1	0.018	0.02	0.1	
Dichlorodifluoromethane	75-71-8	0.013	0.02	0.1	
Ethylbenzene	100-41-4	0.027	0.05	0.1	
Isopropylbenzene	98-82-8	0.026	0.05	0.1	
m & p-Xylene	179601-23-1	0.06	0.1	0.2	
Methyl acetate	79-20-9	0.1	0.1	0.2	
Methyl tert-butyl ether	1634-04-4	0.025	0.05	0.1	
Methylcyclohexane	108-87-2	0.02	0.02	0.1	
Methylene chloride	75-09-2	0.2	0.2	1	
o-Xylene	95-47-6	0.026	0.05	0.1	
Styrene	100-42-5	0.024	0.05	0.1	
Tetrachloroethene	127-18-4	0.01	0.02	0.1	
Toluene	108-88-3	0.011	0.02	0.1	
trans-1,2-Dichloroethene	156-60-5	0.026	0.05	0.1	
trans-1,3-Dichloropropene	10061-02-6	0.014	0.02	0.1	
Trichloroethene	79-01-6	0.02	0.02	0.1	
Trichlorofluoromethane	75-69-4	0.022	0.05	0.2	
Vinyl chloride	75-01-4	0.019	0.02	0.1	
1,2 Dichloroethane-d4	17060-07-0				70 120 % Recovery
Bromofluorobenzene	460-00-4				75 120 % Recovery
d8-Toluene	2037-26-5				85 120 % Recovery
Dibromofluoromethane	1868-53-7				85 115 % Recovery



## STANDARD OPERATING PROCEDURE WC 040 Total Organic Carbon in Soil

Review Date: 01/06/2016

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01/06/2016

Technical Review by:

Date

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01/06/2016

Approved by: Quality Assurance

Date

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## **1.0 Scope and Applicability**

- 1.1 This test method is used to determine Total Organic Carbon (TOC) in soils following the USEPA Lloyd Kahn and SW 846-9060A Methods.
- 1.2 The matrices applicable to this method include soils, sludges, sediments, wastes, and other solid matrices.
- 1.3 Method Detection Limits (MDLs) are determined annually and fall within the range of 250 – 1000 mg/kg.
- 1.4 The current calibration range used for this test method is from 0 – 20 mg of total carbon (see Section 11.2.2).

## **2.0 Summary of Method**

- 2.1 Organic carbon is measured using a carbonaceous analyzer. The instrument converts the organic carbon in a sample to carbon dioxide (CO<sub>2</sub>) by catalytic combustion. The CO<sub>2</sub> formed is then analyzed by an infrared detector. The amount of CO<sub>2</sub> in a sample is directly proportional to the concentration of carbonaceous material in the sample.
- 2.2 Carbonates and bicarbonates are inorganic forms of carbon and must be separated from the total organic carbon value. The carbonate and bicarbonate are removed by treatment with phosphoric acid prior to combustion.

## **3.0 Definitions**

- 3.1 For a list of definitions on many of the terms applicable to this method, see Section 25.6 of the Quality Assurance Manual (QAM).
- 3.2 For a list of common acronyms and abbreviations, see the QAM front matter.

## **4.0 Health and Safety**

- 4.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonable achievable.
- 4.2 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.
- 4.3 The furnace is at 1100°C. Use caution when inserting and removing crucibles from the furnace and when doing any maintenance on the instrument.

4.4 For pollution prevention information, see QAM Appendix 9.

## **5.0 Interferences**

- 5.1 Inorganic sources of carbon (such as carbonates and bicarbonates) represent interference and must either be removed by acidification prior to analysis or accounted for in the final calculation.
- 5.2 Volatile organics in sediments may be lost in the decarbonation step resulting in a low bias.

## **6.0 Equipment and Supplies**

- 6.1 SKALAR, Primacs MCS Solid Sample TOC Module
- 6.2 SKALAR, Formacs CA16 TOC Analyzer
- 6.3 TOC Catalyst: SKALAR catalog no. 2CA10319 or equivalent
- 6.4 Quartz Wool: SKALAR catalog no. 2CA10359 or equivalent.
- 6.5 Ceramic insert tube: SKALAR catalog no. 2SN22254B or equivalent
- 6.6 Quartz crucibles: SKALAR catalog no. 2CS22003 or equivalent
- 6.7 Analytical Balance: Ohaus, Model AP 2500 or equivalent.
- 6.8 Drying Oven: Fisher Isotemp Oven p/n 550-50 or equivalent. Set at 75°C.
- 6.9 Compressed Oxygen: Ultra high purity grade. Airgas or equivalent.
- 6.10 Forceps
- 6.11 For equipment/instrument maintenance, computer hardware and software, and troubleshooting, see QAM Appendix 9.

## **7.0 Reagents & Materials**

- 7.1 Reagent water: Milli Q water >10 mega ohms.
- 7.2 Phosphoric Acid (Fisher catalog number A242SK-212 or equivalent).
- 7.3 25% Phosphoric Acid: Into a 1L volumetric flask, add 250mL concentrated phosphoric acid (7.2) and dilute to volume with reagent water (7.1). Store in the cabinet under the TOC analyzer.
- 7.4 Dextrose: Fisher (Cat. #D16-500 or equivalent).

- 7.5 Potassium Hydrogen Phthalate or an alternate source of Dextrose: E.M. Science (Cat. # DX0145-11).

## 8.0 Sample Handling and Storage

- 8.1 Sampling and storage of samples in glass bottles is preferable but plastic is allowable if it does not contribute to TOC content of sample.
- 8.2 Samples should be stored at 0-6°C until analysis.
- 8.3 Hold time for soil and sludge samples is 28 days. Sediment samples have a hold time of 14 days.

## 9.0 Procedure

**Note:** The following procedure outlines only basic steps for setting up and operating the Primacs MCS instrument. For more detailed information on operating the TOC soil module and TOC4Win MCS software, refer to the SKALAR Primacs MCS user manual.

- 9.1 Start-up:
- 9.1.1 Turn on the mains to the Formacs instrument and the Primacs MCS soil module.
  - 9.1.2 Set the temperature on the Primacs MCS to 1100°C.
  - 9.1.3 Open up the valve on the compressed oxygen. The tank should be set to 30 psi.
  - 9.1.4 Enable the Primacs MCS:
    - 9.1.4.1 Open up the 'HTAccess' software. Once logged in, select "Connection" and then press "Auto-connect." When the analyzer settings table pops up, click "Send settings to analyzer."
    - 9.1.4.2 Put the Formacs analyzer into stand-by mode by clicking the "Settings" tab and then selecting "Stand-By." Make sure that the TC/TN temperature is set to 250°C and the flow is turned off.
    - 9.1.4.3 Click the "View" tab and then select "Control Panel." Click "Enable MCS."
    - 9.1.4.4 Close out of the 'HTAccess' software.
  - 9.1.5 Open up the 'TOC4Win MCS' software. Once logged in, select "Connection" and then press "Auto-connect." When the analyzer settings table pops up, click "Send settings to analyzer."

## 9.2 Sample Preparation:

9.2.1 Weigh approximately 500mg of sample into a quartz crucible.

9.2.1.1 Mix the sample well so that it is homogenous (for some samples, it may be difficult to obtain a representative portion due to the small amount of sample used).

9.2.1.2 If a sample contains a lot of organic material (leaves, twigs, etc.), weigh up a smaller portion of sample.

9.2.1.3 Record the sample weight and place the crucible in a labeled aluminum weigh pan.

9.2.2 Add several drops of 25% phosphoric acid to each sample. Samples that contain inorganic sources of carbon will fizz when acid is added to them. Continue adding acid until the sample no longer fizzes.

9.2.2.1 Acid does not need to be added to Dextrose standards.

9.2.3 Place samples into an oven set to 75°C for approximately 30 minutes.

## 9.3 Run Template Development:

9.3.1 A run template must be created before sample analysis can begin. To make a new template, select the 'template' tab and then click 'new.'

9.3.1.1 For sample analysis, save the template with the date followed by the runs to be analyzed. Example: 021915 112000 112001 112002.

9.3.1.2 For a calibration, save the template with the 'TOC' number for the curve followed by the date analyzed. Example: TOC0001 021915.

9.3.2 There are several key things to look at when creating a new template that are not necessarily default. These items are highlighted in the illustration below:



Template, Edit: C:\nStarch\TOC\SOIL\TEMPLATE\011812 81843.tdb

Position	Identification	Type	Range	Injections	Sample Weight (mg)	TC Weight (mg)	IC Weight (mg)
1	BLANK	Unknown	High	<input type="checkbox"/> TOC <input checked="" type="checkbox"/> TC <input type="checkbox"/> IC	1	1	1
2	CCV	Unknown	High	<input type="checkbox"/> TOC <input checked="" type="checkbox"/> TC <input type="checkbox"/> IC	30.7	1	1
3	LCSS	Unknown	High	<input type="checkbox"/> TOC <input checked="" type="checkbox"/> TC <input type="checkbox"/> IC	32.4	1	1
4	MBS	Unknown	High	<input type="checkbox"/> TOC <input checked="" type="checkbox"/> TC <input type="checkbox"/> IC	500	1	1
5	115946	Unknown	High	<input type="checkbox"/> TOC <input checked="" type="checkbox"/> TC <input type="checkbox"/> IC	1	1	1

No Of Positions: 15    Add Pos    Del Pos    Get Weight TC    Get Weight IC    Renumber    Print

Element: Carbon    Integration Time: 150 s

Sample Time TC: 300 s    Concentration of Standards TC: 40 %  
Sample Time IC: 250 s    Concentration of Standards IC: 11.99 %

Analyser Type: Primacs SLC

Cancel    OK

- 9.3.2.1 Make sure that you enter the weights of your samples and standards in the 'TC Weight' cell.
  - 9.3.2.2 For the analysis of TOC samples, make sure that the 'TC' box is checked.
  - 9.3.2.3 Make sure that the Range you have selected is the same as it was during the last calibration (for soils, we typically use a 'high-range' calibration).
  - 9.3.2.4 Make sure that the 'Integration Time' is the same as it was on the calibration being used.
  - 9.3.2.5 Enter in the "Concentration of Standards." For Dextrose, the carbon concentration is 40%.
- 9.3.3 Add positions to the template that correspond to the QC or samples to be analyzed. The acceptance criteria and the frequency of QC (CCV's, LCS's, MB's, Dup's, etc.) can be found in Table 1 of this SOP.



#### 9.4 Sample Analysis:

- 9.4.1 Create a new analysis run by clicking the "Analysis" tab and then selecting "New."
  - 9.4.1.1 Save the analysis the same way as a template (e.g., 021915 112000 112001 112002).
  - 9.4.1.2 Select the template to be used for the sample analysis (this is typically identical to the analysis run).
- 9.4.2 Select the curve that will be used for sample analysis by clicking "Calibration Curves" in the Results window and then press the "Add Curve" button.
- 9.4.3 Zero-out the baseline by clicking "AutoZero Carbon" in the Graph Peaks window. Be sure that the baseline is stable before starting sample analysis.
- 9.4.4 In the "Analysis Info" text box on the Analysis window, type in the 'W' numbers that correspond to the standards being used for analysis.
- 9.4.5 Click "Start Analysis" when ready to begin analyzing samples.
- 9.4.6 Add samples to the TOC soil module by following the procedure outlined in the Primacs MCS user manual.

#### 9.5 Shut-down and Data Export:

- 9.5.1 When the analysis of all samples and QC is complete and the data have been reviewed, export the results to LIMS. This is done by clicking the "Export Results" button on the Analysis window. Each run must be exported individually. The following table illustrates the proper export layout:

**Export Layout**

☒ **General Info**

- ☒ Analysis Name
- ☒ Template Name
- ☒ Operator
- ☒ Date/Time
- ☐ Start Oven Temp
- ☐ Start Cooler Temp
- ☐ Version
- ☒ Info
- ☐ Method

☐ **Template Info**

- ☐ Element
- ☐ Integration Time
- ☐ Samples
- ☐ Extra Samples
- ☐ Max. CV
- ☐ Sample Time
- ☐ Wash Time
- ☐ Start Ignore Time

☒ **Sample Values**

- ☒ Injection Order
- ☐ Position
- ☒ Type
- ☒ Identification
- ☐ Dilution Factor
- ☐ Range
- ☒ Result TC
- ☐ Result TOC
- ☐ Result NPOC
- ☐ Result IC
- ☐ Result TKN
- ☐ Result TN
- ☐ Result NH
- ☐ Result POC
- ☐ Average Area
- ☐ CV
- ☐ Sample Weight

☒ **Single Values**

- ☐ Concentration
- ☐ Area
- ☐ Link to Curve
- ☐ Oven Temp
- ☐ Cooler Temp
- ☐ Edited
- ☐ Selected
- ☒ Injection Time

☐ **Calibration**

- ☐ General Info

☐ **Type of Samples**

- ☐ Standards
- ☒ Unknowns
- ☐ Quality Samples

**Exit**

- 9.5.2 Manually set the temperature on the Primacs MCS to 40°C. Allow the instrument to cool off with flow.
- 9.5.3 When the instrument is cool, close the valve on the compressed oxygen tank.
- 9.5.4 Turn off the mains to the Primacs MCS and the Formacs.
- 9.5.5 Close down the TOC4Win MCS software.

## 10.0 Calculations

10.1 Dry weight Concentration (mg/kg) = A/B

Where A = instrument reading for sample (mg/kg)  
B = % solids as a decimal

10.2 %RSD = Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$



## 11.0 Calibration & Standardization

- 11.1 To facilitate appropriate peak response and sensitivity, the entire operating system must be correctly set up and maintained before calibration and sample analyses can occur. Using the proper settings and programming will greatly increase the likelihood that calibrations will be acceptable. The manufacturer's recommended settings can be altered to optimize peak shape, reproducibility, and response.

The following table illustrates the settings being utilized currently:

Curve Order	Analyser Type	Miscellaneous	Stand-By
Analysar Settings	Detection Settings	Alarm Settings	
General Primacs SLC			
<b>PrimacsSLC Analyser Settings</b>			
Default Integration Time	150 s		
Default SampleTime TC	300 s		
Default SampleTime IC	250 s		
Default Conc Standard TC	40 %		
Default Conc Standard IC	11.99 %		
✓ TC Temp	250 °C		
✓ IC Temp	50 °C		
✓ TC Sampling Time	300 s		
✓ IC Sampling Time	240 s		
✓ Acid to Cup	600 count(s) * 50 µl		
Send Settings to Analyzer		Restore Defaults	
Reset All		Save Settings	
OK		Cancel	Apply

- 11.2 Calibration Standards – Calibration standards are prepared at a minimum of three concentration levels (although seven levels are currently being used) and are prepared by weighing various amounts of the CCV/Calib. Dextrose standard (Dextrose is 40% carbon). One of the concentration levels shall be at a concentration near, but above, the detection limit and at or below the reporting limit.

The remaining concentration levels shall correspond to the expected range of concentrations found in real samples. The current calibration range for soils is 2mg to 20mg carbon (a 'zero' point is also included in the calibration).

11.2.1 Calibration standards are plotted on a curve by the instrument's computer software. Procedures for programming the calibration are outlined in the reference manuals supplied with the Primacs MCS instrument. The plotted curves must have a correlation coefficient (r) of 0.995 or better in order for the curve to be considered valid. Calibration standards are not static and can be altered as long as linearity can still be demonstrated. It is not allowed to remove any internal curve points in order to make a curve acceptable; however, it is acceptable to reanalyze a curve point if initially unacceptable or to remove the highest point if a loss in linearity is demonstrated.

11.2.2 The following table outlines the preparation of a seven level calibration curve for soil samples:

CCV/Calib. Dextrose (mg)*	Total Carbon (mg)
0	0
5	2
10	4
20	8
30	12
40	16
50	20

\*These are approximate weights.

- 11.3 A second source ICV is analyzed immediately following a successful calibration. An ICV is prepared by weighing approximately 10mg of ICV Dextrose. The acceptance criteria for the ICV are outlined in Table 1 of this SOP.
- 11.4 An ICB is also analyzed when calibrating the instrument. An ICB is prepared by simply analyzing a blank quartz crucible. The acceptance criteria for the ICB are outlined in Table 1 of this SOP.

## 12.0 Quality Control

- 12.1 This SOP is designed to follow a variety of different projects and programs requirements. Table 1 is designed to illustrate the control steps and provisions required to adequately producing acceptable data.
- 12.2 Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.
- 12.3 Program Specific Limits: Samples analyzed under the guidance of certain programs; such as the Department of Defense Quality Systems Manual (DoD/QSM) or Louisville Chemistry Guidance (LCG), require their own specified limits. For these samples follow the limits specified in the manuals for that program.

12.4 Per QSM 5.0, LOD and LOQ checks must be analyzed on a quarterly basis (or once per analytical batch for infrequently performed analyses).

12.4.1 LOD checks should be spiked at a concentration at least two times higher than the calculated MDL.

12.4.2 In the absence of set QSM limits, in-house LCS limits are used for acceptable recovery criteria for LOQ checks.

12.5 Method Performance:

12.5.1 Certified standard solutions and chemicals, properly used instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

12.5.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4-5 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (AR) and the relative standard deviation (RSD) of the recoveries for each analyte. In the absence of specific criteria found in the EPA methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated.

12.5.3 Proper instrument maintenance is another means to ensure adequate method performance. Refer the Shimadzu TOC 500A Instruction Manual or the Shimadzu Solid Sample Module Instruction Manual as needed.

### **13.0 Data Assessment & Acceptance Criteria for QC Measures**

13.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. The analyst will review the data to ensure QC is acceptable and that exceedances are addressed. Acceptable data is then entered into the LIMS system.

13.2 After data has been entered into LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist (FWC40-01) for data review guidance.

13.3 Once the analyst has reviewed and approved the data, it is given to a peer or supervisor for review.

13.4 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.



- 13.5 A paper hard copy of the data is then filed or archived. The package includes the checklist, the sequence run log, and a copy of the bench sheet (if applicable), the LIMS run log, and verification of calibration data.

#### **14.0 Corrective Measures for Handling Out-of-Control Data**

See QAM Appendix 9.

#### **15.0 Contingencies for Handling Out-of-Control or Unacceptable Data**

See QAM Appendix 9.

#### **16.0 Data Records Management**

- 16.1 Records are stored for a minimum of 5 years in accordance with the Quality Manual.

- 16.2 See SOP QA 003 for specifics on document control.

#### **17.0 Waste Management**

See QAM Appendix 9.

#### **18.0 References**

- 18.1 Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method), USEPA, July 27, 1988.
- 18.2 US EPA, SW-846, Method 9060A, Revision 1, November 2004.
- 18.3 SKALAR Formacs TOC/TN Analyzer User Manual, October 2009.
- 18.4 SKALAR Primacs MCS TOC add-on module User Manual, January 2011.
- 18.5 CT Laboratories Quality Manual, current revision.
- 18.6 Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.
- 18.7 National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.
- 18.8 ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

## 19.0 Appendices

**Table 1**  
**Summary of Quality Control Requirements**

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action
4 point curve (3 standards and a blank)	Initially and as needed	$r \geq 0.995$ for each regression line	Repeat until acceptable
Initial calibration verification (ICV)	Second source standard run after each ICAL and daily prior to sample analysis	%R: 90-110%	Reanalyze ICV standard, if ICAL
Initial calibration blank (ICB)	After each ICV, prior to sample analysis	< RL	Remake and reanalyze CB investigate and correct prot than 20 X's the ICB with a 'Y' flag
Continuing calibration verification (CCV)	Daily, prior to sample analysis, after every 10 analyses, and at end of run	%R: 90-110%	Remake and reanalyze CC investigate and correct prot samples after last acceptable
Continuing calibration blank (CCB)	After each CCV	<RL or <20 times less than the lowest sample result	Remake and reanalyze CB investigate and correct prot samples after the last acceptable less than 20 X's the CCB with a 'Y' flag
Laboratory Control Standard (LCS)	Analyzed with each batch of samples (or at project/program specified frequencies)	With in-house limits Default 80-120 %R.) or within project/program specified limits	Remake and reanalyze LCS if unacceptable investigate and correct prot Reanalyze all samples associated with unacceptable reanalysis is not possible samples are 'B' qualified.
Method Blank (MB)	Analyzed with each batch of samples (or at project/program specified frequencies)	< MDL or project/program specified limit. < 1/2 RL for DoD-QSM	If unacceptable, all associated samples are 'B' qualified detects $\leq 20$ times ( or project/program specified limit) and are greater than 1/2 RL for DoD-QSM reanalyzed or 'B' qualified
Matrix Replicate (DUP)	Sample + 3 reps / 20 samples per solids matrix or at project / program frequencies	% RSD within in house limits (Default $\pm 30\%$ ) or project/program specified limits	Qualify results with 'Y' flag
Capability demonstration sample (IDC)	Four (4) prepared samples analyzed one time prior to any sample analyses and one blind sample	Within in house limits. Default 70-130% Recovery $\pm 20\%$ RSD or within project/program specified limits	Repeat until acceptable

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**Table 2**  
**TOC in Soil Data Review Checklist (FWC40-01 Example)**

		Method: Total Organic Carbon: Lloyd Kahn Method / 9060A	Independent Data Review Checklist		
LIMS Run #(s)	Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	

**Instructions:** Complete one checklist per analytical sequence. Enter the appropriate response for each question.

Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

		Analyst Review		Independent Review		(Indicate re- sponse)
		Yes	No	Yes	No	
1. Were the samples acidified prior to analysis and analyzed within hold time?	No effervescence, analyzed within 28 days					If No: Qual
2. Was the calibration curve performed using the required number of standards?	Minimum of 3 standards and a blank					If No: Recs standards
3. Was the correlation coefficient acceptable?	$r \geq .995$					If No: Recs
4. Were the ICV and ICB analyzed?	---					If No: Anal analyses
5. Was the ICV result acceptable?	90 - 110% Rec. or contract/program specific					If No: Rear address pro
6. Was the ICB result acceptable?	< LOD or contract/program specific					If No: addr reanalyze
7. Was an LCS & MB run with each batch of samples?	One each per analytical batch or contract/program specific frequency					If No: Rear appropriate
8. Was the LCS recovery acceptable?	In-house derived or contract/program specific limits					If No: Rear acceptable affected sam
9. Were the MB results acceptable?	< LOD or project/program specific (<1/2 RL for DOD-QSM)					If No: Rear acceptable affected sam
10. Were the CCV's analyzed at the required frequency?	Beginning, after every 10 sample, & at the end of a sequence					If No: Rear appropriate
11. Were the CCB's analyzed at the required frequency?	After each CCV (unless MB analyzed after CCV)					If No: Rear appropriate
12. Were the CCV recoveries acceptable?	90 - 110% Rec. or project/program specific					If No: Rear

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13. Were the OCE results acceptable?	< LOD or project/ program specific					If No: Rem acceptable affected sa
14. Were all positive results that were reported within the calibration curve?	---					If No: Wei reanalyze.
15. Were the appropriate number of samples analyzed in quadruplicate?	1 / 20 of the same matrix or project/ program specific					If No: Prep number of
16. Was the %RSD on the replicated sample acceptable?	≤ 30%					If No: Qua (Y)
20. Are all samples on the job lists accounted for?	---					If No: Ana
21. Is the standards prep log numbers noted on the analytical report?	---					If No: Doc calibration
22. Were post analysis corrections addressed and/or the Audit Trail function turned on (if available)?	Correction should be initialed, dated, and reason given. Audit trail must be on (if available)					If No: Initi changes or
23. Were non-matrix related nonconformities (if applicable) documented in the NCR spreadsheet?	---					If No: Ente NCR sprea

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Table 3  
TOC % RSD Template (FWC40-02 Example)

Total Organic Carbon (TOC)

Analyst:  
Date:  
Run #:  
Sample #:

Matrix = Solids.

% Standard Deviation Calculation: % Std. Dev. = Std Dev./Mean X 100

Sample Result	Dup. 1	Dup. 2	Dup. 3	Mean	Std. Dev.	% Std. Dev.
				0.00	#DIV/0!	#DIV/0!

Limits: 30%



Table 4  
TOC Soil Bench Sheet (FWC40-03 Example)

TOC SOIL BENCHSHEET			
<b>Analyst:</b>		<b>Method:</b>	Lloyd Kahn
<b>Run #(s):</b>		<b>Balance:</b>	WCB01
<b>Date:</b>		<b>CCV Std.:</b>	W23772
<b>Start Time:</b>		<b>LCS Std.:</b>	W23192

	Sample ID	Weight (mg)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		
24		
25		
26		
27		
28		
29		
30		

**Stop Time:**



Revision Number	Description of Changes	Date
2	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	03/12/2014
2.1	Document was reviewed, re-formatted, and updated for QSM 5.0.	02/19/2015
2.2	Removed the Final Concentration column in section 11.2.2 and changed the MDL concentration range in section 1.3.	01/06/2016

This addendum defines project-specific conditions that will be used in the analysis of samples associated with the Remedial Investigation/Feasibility study at the Behr Dayton Thermal System Plume site, Montgomery Co., Dayton, OH. All other conditions and language applies from SOP WC 040.

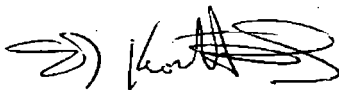
The current Method Detection Limit (MDL) for the analysis to be used in this project is 301 mg/kg.

The calibration range for the test method to be used in this project is from 0 to 12 mg total carbon (0 to 24000 mg/kg of carbon).

The following table outlines the preparation of the seven level-calibration curve to be used in this project:

Amount of Dextrose (mg)*	Total Carbon (mg)	Total Carbon (mg/Kg)
0	0	0
2	0.8	1600
5	2	4000
10	4	8000
15	6	12000
20	8	16000
25	10	20000
30	12	24000

\*These are approximate weights of dextrose to be used the calibration. Actual weights are used in the calculation of the calibration curve.



01/08/2016

Technical Review of Addendum by:

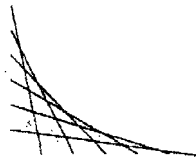
Date



01/08/2016

Approved of Addendum by: Quality Assurance

Date



## STANDARD OPERATING PROCEDURE

### GT 007 Soil Bulk Density- Core Method

Review Date: 11/19/2015

11/19/2015

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Technical Review by:

Date

11/19/2015

---

Approved by: Quality Assurance

Date



**1.0 Identification of the Test Method**

- 1.1 This procedure is used for the analysis of Bulk Density following Methods of Soil Analysis. Part 1. Physical and Mineralogical Properties. 2<sup>nd</sup> ed.

**2.0 Applicable Matrix or Matrices**

- 2.1 This method is applicable to soils, sludges, wastes, and sediments.

**3.0 Detection Limits (n/a)**

- 3.1 Not applicable

**4.0 Scope and Application**

- 4.1 This method is used for determination of soil bulk density by the core method, and is applicable to most types of solid samples.

**5.0 Method Summary**

- 5.1 Soil bulk density is the ratio of the mass of dry solids to the bulk volume of the soil. The determination usually consists of drying and weighing a known volume of a soil sample. The core method is one method for determining bulk density that can be used with either re-packed or natural cores.

**6.0 Definitions**

- 6.1 Reagent Water: Carbon Dioxide (CO<sub>2</sub>) free deionized water (D.I.), >10 mega ohm.  
6.2 Duplicate Analysis: Two aliquots of a given sample are analyzed. The relative percent difference (RPD) is then determined from the two results and compared to the lab control limits for that particular matrix.

**7.0 Interferences**

- 7.1 Improperly core samples that may dissolve or otherwise breakdown during testing are not amenable to this procedure.

**8.0 Safety**

- 8.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to possibly hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems (see CTI Health and Safety Manual).

**9.0 Equipment and Supplies**

- 9.1 Sub-corer (30-mL syringe), BD Medical, Cat. # 309650 or equivalent  
9.2 Top loading balance, Denver Instrument Co. Model XD-2200 or equivalent.  
9.3 Aluminum weighing pan. Fisher Scientific, Cat.# 08-732-103 or equivalent.  
9.4 Drying oven at approximately 103-105°C, Fisher Scientific, Isotemp 500 series or equivalent.

**10.0 Reagents and Materials**

- 10.1 Not Applicable

**11.0 Sample Preservation & Storage**

- 11.1 No preservation of the sample is required but the sample container must remain sealed.  
11.2 Samples are stored cooled to 0 - 6° C until analysis.

## 12.0 Quality Control

- 12.1 This SOP is designed to follow a variety of different projects and programs requirements. Table 1 is designed to illustrate the control steps and provisions required to adequately producing acceptable data.
- 12.2 Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.
- 12.3 Program Specific Limits: Samples analyzed under the guidance of certain programs; such as the Department of Defense Quality Systems Manual (DoD/QSM) or Louisville Chemistry Guidance (LCG), require their own specified limits. For these samples follow the limits specified in the manuals for that program.

## 13.0 Calibration and Standardization

- 13.1 Not applicable for Bulk Density
- 13.2 See SOP (CL-Top Loading Balance Calibration) for balance calibration procedure.

## 14.0 Procedure

- 14.1 Soil samples are typically received in a 1-in or 3-in diameter Shelby tube. Re-packing of un-consolidated samples for bulk density is not recommended but can be performed if the volume of the original soil sample is known.
- 14.2 In order to perform the bulk density, a sub-core is removed with a modified syringe. Care should be taken so as not to greatly modify the soil structure during sub-sampling. Gravel or rocks in the sample may necessitate making repeated attempts at obtaining a representative sub-core. Record the volume of soil in the syringe and extrude the soil into a pre-weighed aluminum, weighing pan. Weigh the wet soil to the nearest 0.01 g and record the weight on the data form. Next, place the pan in the drying oven for a minimum of 4 hours.
- 14.3 Re-weigh the dried soil to nearest 0.01 g and record the dry weight.

## 15.0 Calculations

- 15.1 Calculate the soil Bulk Density using the following formula:

$$\frac{\text{Dry weight of soil}}{\text{Volume of soil}} = \text{gTS/cm}^3$$

- 15.2 Calculate the relative percent difference as follows:

$$\%RPD = \frac{(\text{Orig} - \text{Dup}) \times 100}{(\text{Orig} + \text{Dup})/2}$$

Where:            Orig = Original sample result  
                     Dup = Duplicate sample result

## 16.0 Method Performance

- 16.1 Certified standard solutions and chemicals (if applicable), properly used instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

## **17.0 Pollution Prevention**

- 17.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation.
- 17.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## **18.0 Data Assessment & Acceptance Criteria for QC Measures**

- 18.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. The analyst will review the data to ensure QC is acceptable and that exceedances are addressed. Acceptable data is then entered into the LIMS system.
- 18.2 After data has been entered into LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist (Table 3) for data review guidance.
- 18.3 Once the analyst has reviewed and approved the data, it is given to a peer or supervisor for review.
- 18.4 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.
- 18.5 A paper hard copy of the data is then filed or archived. The package includes the checklist, the sequence run log, a copy of the bench sheet, the LIMS run log, and verification of calibration data.

## **19.0 Corrective Measures for Out-of-Control Data**

- 19.1 When data is out of control, a number of corrective actions may need implementing. If the nonconformities involve failing QC within the analytical sequence batch, then reanalysis of samples may eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data may correct the problem. If sample matrix affect or contamination is the reason for out of control data, the instrument may need cleaning and decontamination. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions should be taken for different QC-type failures or nonconformities (Table 1).

## **20.0 Contingencies for Handling Out-of-Control or Unacceptable Data**

- 20.1 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data may be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their affect on data.
  - 20.1.1 The first thing addressed is prevention of producing unacceptable data. When limited sample volume is the issue, the analyst should determine if splitting the sample into lesser volumes or weights is an option. To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises, reanalysis is an option. Analyst error is prevented by a second analyst confirmation and validation. If the initial analyst makes an analysis



error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.

20.1.2 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more samples can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

20.1.2.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

20.1.2.2 In all instances, if results are reported from data that is out of control or unacceptable, that data should be qualified accordingly. Once the client has been notified and he or she instructs us to report the data, flag the data indicating what type of nonconformity has occurred.

20.1.2.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data. The file folder should be labeled as such, indicating that the data is out of control.

20.1.2.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

## **21.0 Waste Management**

21.0 Samples are routinely held for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (WS001 – Laboratory Waste Disposal).

## **22.0 References**

- 22.1 Methods for Chemical Analysis of Water and Wastes, March, 1983 EPA-600/4-79-020, Method 310.1, Editorial revision 1978.
- 22.2 Standard Methods for the Examination of Water and Wastewater, Method 2320B, 18<sup>th</sup> Edition, 1992, 21<sup>st</sup> Edition, 2005. National Environmental Laboratory Accreditation Program (NELAP), Quality Systems, Chapter 5, June, 2000.
- 22.3 National Environmental Laboratory Accreditation Program (NELAP), Quality Systems, Chapter 5, June, 2000. Model Seven Easy pH meter Operating Instructions, Mettler Toledo
- 22.4 GmbH Analytical, 2003, 2004.
- 22.6 Louisville Chemistry Guideline (LCG), US Army Corps of Engineers-Louisville District, June 2002.

- 22.7 Department of Defense, Quality Systems Manual for Environmental Laboratories, DoD Environmental Data Quality Workgroup, Department of Navy, Lead Service, Draft Version 3, December 2004.
- 22.7 Louisville DOD Quality Systems Manual Supplement (LS), US Army Corps of Engineers-Louisville District, March 2007.
- 22.8 Department of Defense, Quality Systems Manual for Environmental Laboratories, DoD Environmental Data Quality Workgroup, Department of Navy, Lead Service, Based on NELAC Voted Revision 5 June 2003, Version 4.1, April 22, 2009.

**Table 1: Summary of Quality Control Requirements**

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Sample Duplicate (DUP)	1 per 20 per matrix (QSM = 1 / 10) or project / program specific.	In-house derived limits Default: RPD $\leq 20$ or project / program specific limits.	Assess and correct problem, then reanalyze, if necessary, "Y" qualify results.

**Table 2: Bulk Density Bench Sheet**

Date:		Analyst:			LIMS Run #:			
Sample Log #	Pan ID #	Pan Tare Weight (g)	Soil Volume (cubic cm)	Wet weight of Soil and Pan (g)	Wet weight of soil (g)	Dry weight of soil and pan (g)	Dry weight of soil (g)	Soil Bulk Density gm TS/cm <sup>3</sup>
					0.00		0.00	#DIV/0!
					0.00		0.00	#DIV/0!
					0.00		0.00	#DIV/0!
					0.00		0.00	#DIV/0!
					0.00		0.00	#DIV/0!
					0.00		0.00	#DIV/0!
					0.00		0.00	#DIV/0!

Calculation: Dry weight of soil / sample volume



**Table 3: Data Validation Check List**

Method: Bulk Density				
	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes ... No

checklist per *analytical run*. Enter the appropriate response for each question. Each “No” response requires an explanation in the require the initiation of a Nonconformance Report.

Comment:	Acceptance Criteria	Analyst Review		Independent Review		Comments:  (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
quired frequency?	---1 / 20 samples of the same matrix (1/10 for QSM) or program/project specific					
ceptable?	≤20% or use program/project specific limits					
ounted for?	---					

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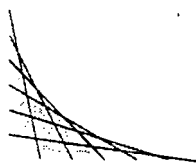


Revision Number	Description of Changes EXAMPLE	Date
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03	Document changed to incorporated administrative requirements of ISO/IEC 17025:2005 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	11/20/15

**SOP #: GT 007**  
**Effective Date: 11/19/15**  
**Revision # 3.0**  
**Page 9 of 9**

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## STANDARD OPERATING PROCEDURE WC 026 Solids, Total

Review Date: 03/19/2015

*Margaret M Lane*

03/19/2015

Technical Review by:

Date

*Colleen Stee*

3/19/2015

Approved by: QA Manager

Date



**1. Identification of Test Method**

- 1.1 This SOP is designed to follow procedures and QC requirements outlined in *Standard Methods for Determination of Water and Wastewater*, Methods 2540B & 2540G and EPA SW-846 method 8000C.

**2. Applicable Matrix or Matrices**

- 2.1 This method is used to determine the percentage of "Total Solids" (TS) in a sample. It is applicable to sludges, soil, surface water, groundwater, and wastewater (both domestic and industrial).
- 2.2 This method can also be used in determining the "% Solids" & "% Moisture" content in solid sample matrices for use in dry weight calculations for other analyses.

**3. Detection Limits**

- 3.1 The Reporting Limits (RL) for this method is 1 mg/L for liquid samples, 0.02% for sludge samples, and 0.2% for soil samples.

**4. Scope & Application**

- 4.1 The result is given as a percentage of the total weight for sludge and soil samples and as mg/L for surface water, groundwater, and wastewater samples.

**5. Method Summary**

- 5.1 A well-mixed sample is evaporated in a weighed dish and dried to a constant weight in an oven at 103-105<sup>0</sup>C. The increase in weight over that of the empty dish represents the total solids.

**6. Definitions**

- 6.1 Duplicate Analysis: Two aliquots of a given sample are analyzed. The difference is then determined from the two results and compared to the laboratory or project specific control limits.
- 6.1.1 Duplicate at least 10 percent of the samples for Total Solids and 5% of the samples for % Solids.

**7. Interferences**

- 7.1 Highly mineralized water, with a significant concentration of calcium, magnesium, chloride and/or sulfate, may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.
- 7.2 Exclude, floating particles or submerged agglomerates of non-homogeneous materials from the sample if it is determined that their inclusion is not desired in the final result.
- 7.3 Because excessive residue in the dish may form a water-trapping crust, limit sample to no more than 200 mg residue.
- 7.4 The determination of total solids in solid and semisolid materials is subject to negative error due to loss of ammonium carbonate and volatile organic matter during drying. Although this is true also for wastewater, the effect tends to be more pronounced with sediments, and especially with sludges and sludge cakes.

The mass of organic matter recovered from sludge and sediment requires a longer ignition time than that specified for wastewaters, effluents, or polluted waters. Carefully observe specified ignition time and temperature to control losses of volatile inorganic salts if these are a problem. Make all weighing of samples quickly because wet samples tend to lose weight by evaporation. After drying or ignition, residues often are very hygroscopic and rapidly absorb moisture from the air.

## **8. Safety**

- 8.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to possibly hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems (see CTI Health and Safety Manual).

## **9. Equipment & Supplies**

- 9.1 Aluminum weighing dishes or porcelain evaporation dish.
- 9.2 Drying oven, capable of 103-105°C (Fisher, Isotemp 500 series or equivalent).
- 9.3 Top Loading balance, capable of weighing to 0.01 g (used for soil samples). Denver Instruments, XD2200 or equivalent.
- 9.4 Analytical balance, capable of weighing to 0.0001 g (used for liquid and sludge samples). Ohaus Voyager Pro or equivalent.
- 9.5 Scoop or spatula.
- 9.6 Wide-bore pipettes.
- 9.7 Desiccator, provided with a desiccant containing a color indicator of moisture. Drierite (Calcium carbonate) or equivalent.

## **10. Reagent & Materials**

- 10.1 There are no special standards or reagents required with this analysis.

## **11. Sample Preservation and Storage**

- 11.1 Samples are collected in resistant-glass or plastic bottles or 4 oz jars, providing that the material in suspension does not adhere to container walls.
- 11.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to <4°C, to minimize microbiological decomposition of solids, is recommended.
  - 11.2.1 Samples are stored in a refrigerator unit until analysis.
  - 11.2.2 Samples analyzed following Standard Methods 2540B & 2540G shall be analyzed within 7 days.

## **12. Quality Control**

- 12.1 Duplicate analysis for Total Solids shall be done for every 10% of samples per matrix. The Relative Percent Difference (RPD) for the duplicates must be less than or equal to the control limits (Table 1). If the result of the replicate exceeds the quality control limit corrective action must take place. Corrective action shall include reanalysis of affected samples or qualifying the results back to the last acceptable quality control check, unless the laboratory determines the sample

results are unaffected (in this case, the rationale must be noted along with the data package).

- 12.2 A duplicate is needed for every 20 samples requiring % Solids/Moisture analyses and also must be within established control limits.
- 12.3 Calibration of the analytical balance prior to use will ensure accurate measurements.
- 12.4 Weighing the sample to constant weight ensures that the sample is not gaining moisture once removed from the oven.

### 13. Calibration & Standardization

- 13.1 Follow manufacturer's instructions to calibrate the balances (See SOP's CC-Ohaus Balance Calibration & CL- Top Loading Balance Calibration for calibration procedures).

### 14. Procedure

- 14.1 If only total solids are requested, heat clean aluminum dishes to 103 – 105° C for 1 hour. If sample also needs total volatile solids, use an evaporating dish that has been ignited at  $500^{\circ} \pm 50^{\circ}\text{C}$  for one hour.
- 14.2 Transfer sample (amount specified below) to a pre weighed dish, and weigh both dish and sample. Record weight.
  - 14.2.1 For soil samples: utilizing the top loading balance, transfer 5-10 g of well mixed sample to a weigh dish, with a scoop or spatula.
  - 14.2.2 For water samples: utilizing the analytical balance, transfer 25-100 ml of well mixed sample to weigh dish, with a pipette. NOTE: If the sample contains suspended solids, it may be necessary to use a wide-bore pipette.
  - 14.2.3 For sludge samples: utilizing the analytical balance, transfer 10-50 g of well mixed sample to dish. NOTE: If the sludge is a liquid, stir to homogenize before transferring aliquot. If the sludge is a solid, pulverize to homogenize the sample before transferring to the weigh dish.
- 14.3 Place the dish into an oven at 103-105°C, dry overnight (at least 8 hrs.).
- 14.4 When drying is complete, the samples may be removed from the oven, cooled in a desiccator, weighed back and reported.
  - 14.4.1 A second reading on a selected sample is taken and must agree with the initial reading within 4% or 50 mg (whichever is less).
  - 14.4.2 If the second reading does not meet criteria place all samples back into the oven for 1 hour and repeat procedure 14.4.1 until acceptable results are obtained.

### 15. Calculations

- 15.1 For solid sample, use the spreadsheet located in H:\INSTRMNT\SOLIDS\TS.
- 15.2 For sludge and water samples, use the spreadsheet located in H:\INSTRMNT\SOLIDS\TSTVS.
- 15.3 Sludge & Soil Samples (%):

$$\text{Total Solids/\% Solids} = \frac{(A - B)}{B} \times 100$$



$$(C - B)$$

$$\% \text{ Moisture} = 100 - \frac{(A - B) \times 100}{(C - B)}$$

where:

A = Dry weight of sample and dish, g

B = Tared weight of dish, g

C = Weight of sample and dish, g

#### 15.4 Water Samples (mg/L):

$$\text{Total Solids (mg/L)} = \frac{(A-B) \times 1000}{\text{Sample volume, ml}}$$

where:

A = weight of dried residue + dish, mg

B = weight of dish, mg

#### 15.5 Precision (%):

##### **Relative percent difference (RPD)**

$$\frac{[\text{sample result} - \text{duplicate sample result}]}{[(\text{sample result} + \text{duplicate sample result}) / 2]} \times 100 = \% \text{RPD}$$

### **16. Method Performance**

- 16.1 Properly used equipment, and analyst experience and expertise are critical elements in producing accurate results. Equipment performance is continually checked and documented in instrument logbooks
- 16.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4-5 laboratory control samples. After analysis, the analyst calculates the relative standard deviation (RSD) between the analyses. In general "Total Solids results shall agree within 5%.
- 16.3 Proper equipment maintenance is another means to ensure adequate method performance.

### **17. Pollution Prevention**

- 17.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation.

### **18. Data Assessment & Acceptance Criteria for QC Measures**

- 18.1 When the preparation of an analytical batch has been completed, the samples are analyzed and prepared for reporting. The analyst will review the data to ensure QC is acceptable and that exceedances are addressed. Acceptable data is then captured into the LIMS system.
- 18.2 After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness.
- 18.3 Once the analyst has reviewed and approved the data, it is given to a peer or supervisor for review.
- 18.4 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.
- 18.5 A paper hard copy of the data is then filed or archived. The package includes any checklists, the sequence run log, the prep batch, and a copy of the bench sheet (FWC26-(2-5)), the LIMS run log, and verification of calibration data.

## **19.0 Corrective Measures for Out-of-Control Data**

- 19.1 When data is out of control, a number of corrective actions may need implementing. If the nonconformities involve failing QC within the analytical sequence batch, then reanalysis of samples may eliminate any out of control data. If the out of control data is the result of equipment malfunctions, then maintenance or repair of the downed equipment followed by re-preparation of affected samples may correct the problem. If sample matrix affect or contamination is the reason for poor data, the analysis instrument may need cleaning and decontamination. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions should be taken for different QC-type failures or nonconformities (Table 1).

## **20.0 Contingencies for Handling Out-of-Control or Unacceptable Data**

- 20.1 Due to limited sample volume, expiration of hold times, downed equipment and instrumentation, and analyst error, the sample data may be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their affect on data.
  - 20.1.1 The first thing addressed is prevention of producing unacceptable data. When limited sample volume is the issue, the analyst should determine if splitting the sample into lesser volumes or weights is an option. To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises, reanalysis is an option. Analyst error is prevented by a second analyst confirmation and validation. If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.
  - 20.1.2 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client

services will inform the client and/or responsible parties. In some instances, more samples can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

- 20.1.2.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.
- 20.1.2.2 In all instances, if results are reported from data that is out of control or unacceptable, that data should be qualified accordingly. Once the client has been notified and he or she instructs us to report the data, flag the data indicating what type of nonconformity has occurred.
- 20.1.2.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data. The file folder should be labeled as such, indicating that the data is out of control.
- 20.1.2.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

## 21.0 Waste Management

- 21.1 Samples are routinely held for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).

## 22.0 REFERENCES

- 22.1 *Standard Methods for Determination of Water and Wastewater*, 18th Edition, 1992, APHA-AWWA-WEF, Method 2540B & 2540G.
- 22.2 USEPA, SW-846, Method 8000C, Rev. 3, March, 2003.
- 22.3 CT Laboratories Quality Manual, current revision.
- 22.4 Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.
- 22.5 National Environmental Laboratory Accreditation Conference (NELAC), 2003. NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, or most recent version.
- 22.6 ISO. 2005. General Requirements for the competence of testing and calibration laboratories. ISO17025.



**Table 1: SUMMARY OF QUALITY CONTROL REQUIREMENTS**

<b>QC Type</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action if Unacceptable</b>
Sample Duplicate (DUP)	1 per 10 samples for TS, 1 per 20 sample for % Solids or % Moisture	In-house derived or client/project specific limits: Default: $RPD \leq 5\%$	Investigate problem, if system precision in control qualify results, if system precision out of control reanalyze entire batch
Constant Weight Sample (Second weighing)	Check one (1) sample per batch	Weigh until constant weight of or until the weights agree within 4% or 50 mg.	Place back in oven and repeat weighing process until constant weight is achieved
Capability demonstration sample (IDC)	Four (4-5) prepared samples analyzed one time prior to any sample analyses	In-house determined criteria for accuracy and precision	Repeat until acceptable



**FWC26-01**  
**Total Solids Data Validation Checklist**

LIMS #:		Method: Total Solids SM2540B&G / Percent Solids SW846-8000C		
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes ... No

**Instructions:** Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Were samples analyzed within hold time?	7 days for TS					
2. Were samples dried overnight?	> 8hours					
3. Were drying start and stop times recorded?	---					
4. Were duplicates analyzed at the appropriate frequency?	1 per 10 TS or 1 per 20 for % Solids					
5. Were the duplicates within acceptable limits?	Within in house QC limits					
6. Are all samples on the job lists accounted for?	---					



**FWC26-02**  
**Total Solids & TVS in Liquids Spreadsheet**

FWC26.27-02 Total Solids & Total Volatile Solids in Liquids										TS Run #	TVS Run #		
Date: _____										Method: SM2540B			
Sample ID#	Dish#	DF	Tare Wt. g (E)	Vol. ml (F)	Dry Wt. g (G)	Ignited Wt. g (H)	Total Solids mg/L	Vol. Solids mg/L	Flags	Comments			
1		1					#DIV/0!	0.00					
2		1					#DIV/0!	0.00					
3		1					#DIV/0!	0.00					
4		1					#DIV/0!	0.00					
5		1					#DIV/0!	0.00					
6		1					#DIV/0!	0.00					
7		1					#DIV/0!	0.00					
8		1					#DIV/0!	0.00					
9		1					#DIV/0!	0.00					
*10		1					#DIV/0!	0.00					
dup 10		1					#DIV/0!	0.00					
* 2nd Reading.			* 2nd Reading.			Average:	#DIV/0!	0.00	*2nd reading must be within 0.5 mg of the 1st reading				
mg Difference			0.00			TS RPD=	#DIV/0!						
						TVS RPD=	0.0%						
11		1					#DIV/0!	0.00					
12		1					#DIV/0!	0.00					
13		1					#DIV/0!	0.00					
14		1					#DIV/0!	0.00					
15		1					#DIV/0!	0.00					
16		1					#DIV/0!	0.00					
17		1					#DIV/0!	0.00					
18		1					#DIV/0!	0.00					
19		1					#DIV/0!	0.00					
*20		1					#DIV/0!	0.00					
dup 20		1					#DIV/0!	0.00					
* 2nd Reading.			* 2nd Reading.			Average:	#DIV/0!	0.00	*2nd reading must be within 0.5 mg of the 1st reading				
mg Difference			0.00			TS RPD=	#DIV/0!						
						TVS RPD=	0.0%						
Balance: Voyager Pro													
Start Time: _____													
Stop Date: _____													
Stop Time: _____													
DF correction calculated in LIMS													
							Calculations TS, mg/L $\frac{(G-E) \times 1000}{(F/1000)}$ TVS, mg/L $\frac{(H-E) \times 1000}{(F/1000)}$						



### FWC26-03 Total Solids (Percent) Spreadsheet

FWC26-03 TOTAL SOLIDS (PERCENT)						LIMS #:
Start Date:	Start Time:				Analyst:	
Sample ID#	Dish#	Tared Weight g (D)	Wet Weight g (E)	Dry Weight g (F)	RESULTS % TOTAL SOLIDS	
1)						0.0%
2)						0.0%
3)						0.0%
4)						0.0%
5)						0.0%
6)						0.0%
7)						0.0%
8)						0.0%
9)						0.0%
10)						0.0%
11)						0.0%
12)						0.0%
13)						0.0%
14)						0.0%
15)						0.0%
16)						0.0%
17)						0.0%
18)						0.0%
19)						0.0%
*20)						0.0%
Dup 20)						0.0%
Dry Weight = Sample + Dish (gms)				* 2nd Reading.		
Wet Weight = Sample + Dish (gms)				Set RPD:		0%
Balance: XD-2200				*mg Difference		0
Stop Date:				Calculations		
Stop Time:				$\% \text{ Total Solids} = ((F-D)/(E-D)) * 100$ $\text{RPD, \%} = \text{Absolute value of } ((\text{Sample-Dup \% TS})/(\text{Average \% TS})) * 100$		
*2nd reading must be within 50mg of the 1st						



# FWC26-04 Total Solids (Percent) & TVS Sludge Spreadsheet

TS TVS Sig Template

FWC26.27-04											
Total Solids (Percent) and Total Volatile Solids						LIMS (%TS) #:	LIMS (TVS) #:				
Start Date	Method: SM2540B			Analyst:							
Sample ID#	Dish#	Tare Wt. g (D)	Sample Wet Wt. g (E)	Dry Wt. g (F)	Total Solids %	Ignited Wt. g (H)	Vol Solids gVS/ gTS	Matrix Type sludge (s) soil (s)		Flags/ Comments	
1					0.00		0.0000				
2					0.00		0.0000				
3					0.00		0.0000				
4					0.00		0.0000				
5					0.00		0.0000				
6					0.00		0.0000				
7					0.00		0.0000				
8					0.00		0.0000				
9					0.00		0.0000				
10					0.00		0.0000				
11					0.00		0.0000				
12					0.00		0.0000				
13					0.00		0.0000				
14					0.00		0.0000				
15					0.00		0.0000				
16					0.00		0.0000				
17					0.00		0.0000				
18					0.00		0.0000				
19					0.00		0.0000				
*20					0.00		0.0000				
Dup 20					0.00		0.0000				
* 2nd Reading,		2nd Reading,		Average:		Average:					
Total Solids	0.00	Vol. Solids	0.0000								
mg Difference	0.00	mg Difference	0.00								
		TS RPD=	0%	TVS RPD=	0%						
						$\%TS = \frac{(E-D) \times 100}{(E-D)}$		$gVS/gTS = \frac{(F-H)}{(F-D)}$			
2nd Reading must be within 50 mg of the 1st		Balance: Voyager Pro									
Start: Time:		Stop Date:		Stop Time:							

%TSTVS1



# FWC26-05

## Total Solids & TVS Percent Moisture Spreadsheet

TS TVS %M Template

FWC26-05											
Total Solids (%) Total Volatile Solids and % Moisture						LIMS #:					
Date:	Method: SM2540B			Analyst:		(% Moisture 7):					
Sample ID#	Dish#	Tare Wt. g (D)	Sample Wet Wt. g (E)	Dry Wt. g (F)	Total Solids % (G)	Ignited Wt. g (H)	Vol Solids gVS: gTS	Matrix Type sludge (sl) soil (so)		% Moisture	Comments
1					0.0%		0.0000			100.0%	
2					0.0%		0.0000			100.0%	
3					0.0%		0.0000			100.0%	
4					0.0%		0.0000			100.0%	
5					0.0%		0.0000			100.0%	
6					0.0%		0.0000			100.0%	
7					0.0%		0.0000			100.0%	
8					0.0%		0.0000			100.0%	
9					0.0%		0.0000			100.0%	
10					0.0%		0.0000			100.0%	
11					0.0%		0.0000			100.0%	
12					0.0%		0.0000			100.0%	
13					0.0%		0.0000			100.0%	
14					0.0%		0.0000			100.0%	
15					0.0%		0.0000			100.0%	
16					0.0%		0.0000			100.0%	
17					0.0%		0.0000			100.0%	
18					0.0%		0.0000			100.0%	
19					0.0%		0.0000			100.0%	
*20					0.0%		0.0000			100.0%	
Dup20					0.0%		0.0000			100.0%	
* 2nd Reading		2nd Reading			TS Average	0.0%		Calculations			
Total Solids	0.00	Vol. Solids	0.00		$\%TS = \frac{(F-D)}{(E-D)} \times 100$ $gVS:gTS = \frac{(F-D)}{(E-D)}$						
mg Difference	0	mg Difference	0								
Balance: Voyager Pro		TS RPD	0.0%	TVS RPD	=DIV/DI						
2nd Reading must be within 50 mg of the 1st Balance: Voyager Pro											
Start: Time:		Stop Date:		Stop Time:				% M = 100% - G			

%TSTVS%M 1



Revision  
Number

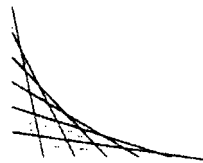
Description of Changes

Date

04	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	04/03/2014

# CT LABORATORIES

*delivering more than data from your environmental analyses*



SOP #: GT 004  
Effective Date: 3/27/15  
Revision #: 3.1  
Page 1 of 9

## STANDARD OPERATING PROCEDURE GT 004 Grain Size

Review Date: 03/12/2015

*Randy Dy*

03/12/2015

Technical Review by:

Date

*Colleen Stuei*

3/27/15

Approved by: Quality Assurance

Date

THIS DOCUMENT IS UNCONTROLLED WHEN PRINTED

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## **1. SCOPE AND APPLICABILITY**

- 1.1. This method is used for determination of particle size of a solid matrix.
- 1.2. Applicable matrices for this procedure include sands and loose top-soil.
- 1.3. This procedure is not intended for sediments or fine clays (these require wet-sieving).

## **2. SUMMARY OF METHOD**

- 2.1. Sample is dried and separated with sieves of different mesh sizes. The sample is passed through the sieves starting with the largest mesh and descending to the smallest in order to determine the percentage of soil passing through each sieve size.

## **3. DEFINITIONS**

- 3.1. % Passing -The fractional equivalent of soil that passes through a known size of a particular sieve starting with an initial weight of sample and subtracting the weight of remaining sample passing through each sieve.

## **4. HEALTH AND SAFETY**

- 4.1. Gloves and protective clothing should be worn to protect against unnecessary exposure to possibly hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.
- 4.2. For pollution prevention information, see QAM Appendix 9.

## **5. INTERFERENCES**

- 5.1. Sample aggregate that is not completely dry.
- 5.2. Sample aggregate that has been oven-dried at too high of a temperature which causes clumping (sample is normally air dried, but oven drying very wet samples to drive off the excess moisture can be done as long as sample is removed before clumping of the sample occurs).

## **6. EQUIPMENT AND SUPPLIES**

- 6.1. Balance; balance sensitive to 0.1 g, Denver Instruments, Model APX-6001 or equivalent

- 6.2. Sieve series, #4, #10, #40, #60, #80, #100 and #200, #230 with a collection pan, Fisher Scientific-USA Standard Test Sieve or equivalent
- 6.3. Mechanical sieve shaker, Humboldt MFG Co.
- 6.4. Aluminum weighing pan. Fisher Scientific, Cat. # 08-732-103 or equivalent
- 6.5. Drying oven at 103-105°C, Fisher Scientific, Isotemp 500 series or equivalent
- 6.6. Pestle and mortar (Porcelain), Fisher (CoorsTek) or equivalent

## **7. REAGENTS AND STANDARDS**

- 7.1. There are no reagents or standards for this method.

## **8. SAMPLE HANDLING AND PRESERVATION**

- 8.1. No preservation of the sample is required. Samples are collected in either 4-oz or 8-oz jars.
- 8.2. Samples are stored cooled to 0 - 6° C until analysis.

## **9. PROCEDURE**

- 9.1. Using a drying pan, thoroughly dry sample by exposing it to room temperature. For best analysis results, use enough sample to ensure at least 100 - 150 g of dried sample.
- 9.2. Break up sample aggregate with a pestle and mortar, being careful not to disintegrate sample. Weigh the sample and record the weight on the Grain Size Bench Sheet (Figure 1).
- 9.3. Place the dry/weighed sample into the sieves stacked in series from largest mesh to smallest, and shake sample on mechanical shaker for five to ten minutes.
- 9.4. After sample is shaken, weigh the portions collected on each sieve and record as weight retained on each of the sieves used.

## **10. CALCULATIONS AND DATA ANALYSIS AND REDUCTION**

Calculate the % Passing through each sieve using the following formula:

Example:

$$\% \text{ Passing \#4} = \frac{(\text{Total weight of Sample}) - (\text{Weight Retained on \#4})}{\text{Total Weight of Sample}} \times 100$$

Note: Calculate each % passing for the remaining sieves used, remembering to subtract the weight retained on each of the previous sieves along with the weight of the sample retained on the sieve of interest.

## **11. CALIBRATION AND STANDARDIZATION**

- 11.1. Not applicable

## **12. QUALITY CONTROL**

- 12.1. This SOP is designed to follow a variety of different project and program requirements. There are no known QC requirements for grain size analysis (i.e. no Duplicate, MS/MSD, or LCS requirements).

## **13. DATA ASSESSMENT/ACCEPTANCE CRITERIA FOR QC MEASURES**

- 13.1. When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. The analyst will review the data and enter the results into the LIMS system. See checklist (Table 2 (Form # GT4-02)) for data review guidance.
- 13.2. After data has been entered into LIMS, it is reviewed by the analyst for accuracy and completeness. Once the analyst has reviewed and approved the data, it is given to a peer or supervisor for review.
- 13.3. After second review approves the data, the reviewer changes the data to "validated" status in LIMS.
- 13.4. A paper hard copy of the data is then filed or archived. The package includes the checklist, a copy of the bench sheet, and the LIMS run log.

## **14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA**

- 14.1. Not applicable

## **15. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA**

- 15.1. Not applicable

## **16. DATA RECORDS MANAGEMENT**



- 16.1. Records are stored for a minimum of 5 years in accordance with the Quality Manual.
- 16.2. See SOP QA 003 for specifics on document control.

## **17.WASTE MANAGEMENT**

See QAM Appendix 9.

## **18.REFERENCES**

- 18.1. ASTM C136-84a and ASTM C3390, Modified.
- 18.2. National Environmental Laboratory Accreditation Program (NELAP), Quality Systems, Chapter 5, June, 2003.
- 18.3. Model Seven Easy pH meter Operating Instructions, Mettler Toledo GmbH Analytical, 2003, 2004.
- 18.4. Louisville Chemistry Guideline (LCG), US Army Corps of Engineers-Louisville District, June 2002.
- 18.5. Louisville DOD Quality Systems Manual Supplement (LS), US Army Corps of Engineers-Louisville District, March 2007.
- 18.6. Department of Defense, Quality Systems Manual for Environmental Laboratories, DoD Environmental Data Quality Workgroup, Department of Navy, Lead Service, Based on NELAC Voted Revision 5 June 2003, Version 5.0, April 22, 2009.
- 18.7. National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 ore most recent revision.
- 18.8. ISO. 2005. General Requirements for the competence of testing and calibration laboratories. ISO/IEC17025:2005.
- 18.9. CT Laboratories Quality Manual, current revision.

## 19.FIGURES

Figure 1. Sieve Analysis Bench Sheet (FGT4-01 Example)

FORM #: FGT4-01  
Rev. #: 1.0  
Effective Date: 05/19/2014  
Page 1 of 7

**GRAIN SIZE SIEVE ANALYSIS**

PAGE 1 Run #:

ANALYSIS DATE/TIME: INITIAL WEIGHT(g):  
ANALYST: #DIV/0!  
SAMPLE ID#: #DIV/0!

**Sample 1** Weight of Soil in Pan (g)

WEIGHT RETAINED ON #4	%PASSING#4	% Passing
WEIGHT RETAINED ON #10	%PASSING#10	#DIV/0!
WEIGHT RETAINED ON #20	%PASSING#20	#DIV/0!
WEIGHT RETAINED ON #40	%PASSING#40	#DIV/0!
WEIGHT RETAINED ON #60	%PASSING#60	#DIV/0!
WEIGHT RETAINED ON #80	%PASSING#80	#DIV/0!
WEIGHT RETAINED ON #100	%PASSING#100	#DIV/0!
WEIGHT RETAINED ON #200	%PASSING#200	#DIV/0!
WEIGHT RETAINED ON #230	%PASSING#230	#DIV/0!

ANALYSIS DATE/TIME: INITIAL WEIGHT(g):  
ANALYST: #DIV/0!  
SAMPLE ID#: #DIV/0!

**Sample 2** Weight of Soil in Pan (g)

WEIGHT RETAINED ON #4	%PASSING#4	% Passing
WEIGHT RETAINED ON #10	%PASSING#10	#DIV/0!
WEIGHT RETAINED ON #20	%PASSING#20	#DIV/0!
WEIGHT RETAINED ON #40	%PASSING#40	#DIV/0!
WEIGHT RETAINED ON #60	%PASSING#60	#DIV/0!
WEIGHT RETAINED ON #80	%PASSING#80	#DIV/0!
WEIGHT RETAINED ON #100	%PASSING#100	#DIV/0!
WEIGHT RETAINED ON #200	%PASSING#200	#DIV/0!
WEIGHT RETAINED ON #230	%PASSING#230	#DIV/0!

ANALYSIS DATE/TIME: INITIAL WEIGHT(g):  
ANALYST: #DIV/0!  
SAMPLE ID#: #DIV/0!

**Sample 3** Weight of Soil in Pan (g)

WEIGHT RETAINED ON #4	%PASSING#4	% Passing
WEIGHT RETAINED ON #10	%PASSING#10	#DIV/0!
WEIGHT RETAINED ON #20	%PASSING#20	#DIV/0!
WEIGHT RETAINED ON #40	%PASSING#40	#DIV/0!
WEIGHT RETAINED ON #60	%PASSING#60	#DIV/0!
WEIGHT RETAINED ON #80	%PASSING#80	#DIV/0!
WEIGHT RETAINED ON #100	%PASSING#100	#DIV/0!
WEIGHT RETAINED ON #200	%PASSING#200	#DIV/0!
WEIGHT RETAINED ON #230	%PASSING#230	#DIV/0!



Figure 2. Grain Size Checklist (FGT4-02 Example)

FORM #: FGT4-02  
Rev. #: 1.0  
Effective Date: 10/13/2014

LIMS #:		Method: Grain Size	
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review
			Approved
			Yes

**Instructions:** Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review	Review	Comments: (indicate reference to an attachment if necessary)
		Yes	No			
1. Was enough sample provided to perform analysis?	Minimum of 100-150 g of dried sample.					
2. Are all samples on the job lists accounted for?	---					
3. Were non-conformities (if applicable) documented in the NCR spreadsheet?	---					If No, enter nonconformities into the NCR spreadsheet before data review/validation.

Revision Number	Description of Changes	Date
3.1	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document. Document was re-formatted.	03/12/2015